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Plasma FIB Spin Milling for Large Volume Serial Sectioning Tomography

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Many material systems need to be analysed in three dimensions (3D) to understand a true nature of connectivity of phases, porous networks and complex shapes. Several tools are available for 3D characterization e.g. X-ray computed Tomography (CT) [1], Serial Sectioning Ga^+ FIB-SEM Tomography (SST) [2, 3], Xe^+ Plasma FIB-SEM SST [4, 5], tri-beam SST [6, 7], transmission electron microscopy [8] and Atom Probe [9]. Figure 1 demonstrates their complementarity in terms of the lengthscales they can access.

More than a decade ago the advent of dual beam focused ion beam (FIB-SEM) using Ga^+ ions has provided a means of accessing regions of interest of 50 μm dimensions both for site specific TEM and SST in the SEM with slice thicknesses down to 10 nm. This destructive enabled 3D imaging of structure, grain structure (via EBSD) and chemistry (by EDS). Recently the limits on volumes accessible through the Ga^+ Ion FIB have been suppressed by emergence of Plasma Xe^+ FIB (PFIB) instruments [4, 5] and tri-beam (fs-laser PFIB) [6, 7] and currently the multi-ion species Thermo Scientific™ Helios™ Hydra™ dualbeam PFIB-SEM allowing access of 100's – 1000's of microns[3]. PFIB having similar nano-scale capabilities of Ga^+ FIB greatly increases both the size of the volume that can be examined and the depth from which the regions of interest identified by x-ray CT can be recovered for higher scale investigation within a correlative tomography framework [1].

There are many questions in materials science and life science that require analysis across length-scales up to many 100's – 1000's of microns. These relating to grain microtextures, grain neighbourhoods, grain boundaries, voids, grain boundary precipitates, inclusions and cracks are of importance for material science. While unravelling complex 3D architecture of cells and tissues in their natural context is crucial for the structure function correlation in biological systems.

In this work we present the first examination of the capabilities of PFIB-SEM for machining and layer removal at glancing angles (0.5 – 1 deg) to the sample surface, so called spin milling (SM). We show that the Helios™ dualbeam Xe^+ PFIB using various currents (60 nA – 2500 nA) can mill areas in excess of $1000 \times 1000\ \mu m^2$ achieving repeatable removal of layers dozens of nm thick, as measured by the cross-holes method proposed in [10]. The performance of the PFIB SM spin milling is demonstrated through the study of Zr-based bulk metallic glass containing crystalline dendrites BMGMC, see Fig. 2, (previously serial sectioned using Xe^+ PFIB and Auto Slice & View software in [4]).

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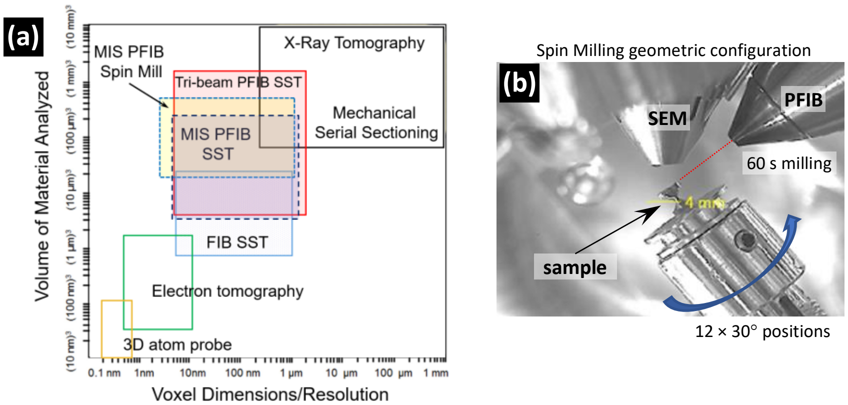

Figure 1. (a) Comparison of 3D tomography methods. MIS – multi-ion species Hydra^{TM} PFIB; SST – serial sectioning tomography; Tri-beam is fs-laser beam plasma FIB-SEM. **(b)** the spin mill setup.

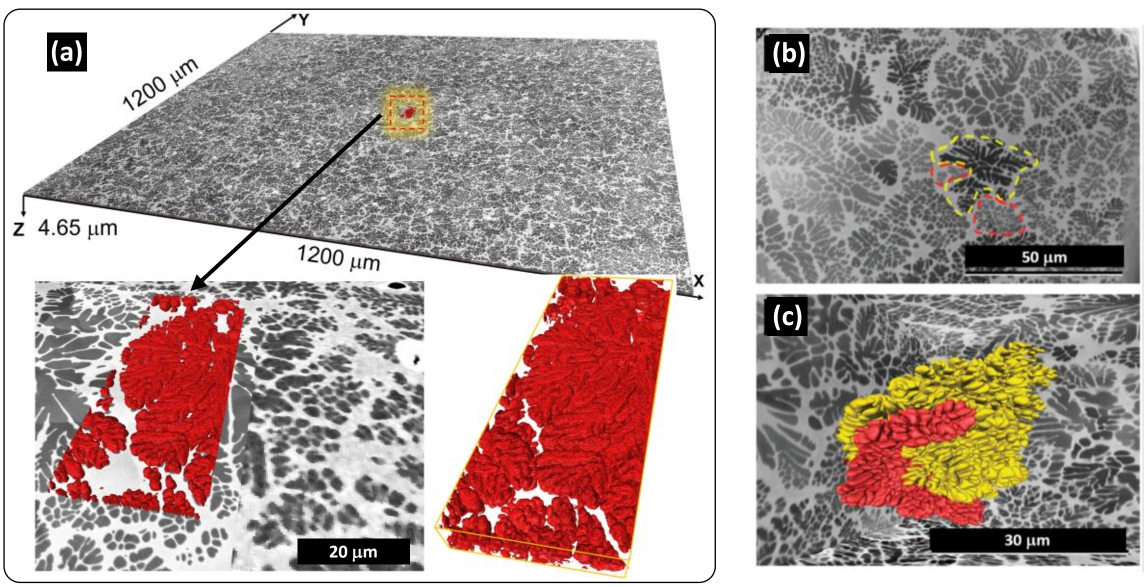

Figure 2. Avizo 3D rendering of the BMGMC serial sectioned by PFIB spin milling at 0.5° glancing angle, 30 kV / 500 nA, 925×50 µm^2 milling area. **(a)** Shows the BMGMC material volume of ~6.7×10^{-3} mm^3. Data contains 260 sections, total milling time ~52h; inset shows SEMs collected with different resolution and detail of the region in center location in a) and 3D rendering of a crystalline dendrite. **(b)** BMGMC serial sectioned by PFIB at 30 kV / 60 nA (700 sections, ~24h total milling time), ~0.5×10^{-3} mm^3, after [4]. SEM image of a single PFIB-prepared slice of the BMG showing the dendrites. Two interlocked red and yellow grains are highlighted in outline. **(c)** Rendering showing two dendrites of which b) shows only a section through.

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